Comparison of Photoacoustic Methods to Loss-on-Ignition and Foam Index Tests in Fly Ash Evaluations

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The traditional method of measuring the mass of carbon in ash, known as loss-on-ignition (LOI), involves burning out the carbon. The technique is not totally satisfactory and efforts have been made to make it faster, more convenient, and more accurate. In one important application, the substitution of ash for portland cement, the mass of carbon is not as important as its tendency to absorb surfactant added to cement mixes to improve mechanical properties. The Foam Index has been developed in recent years as a relative simple wet chemistry test of the suitability of particular coal ashes in cement mixes. In essence, this test measures the effective surface area of carbon in an ash sample.

We are investigating the usefulness of photoacoustic (PA) technology to simplify measurement of either the volume (mass) of carbon or the surface area of carbon in an ash sample. The PA effect is the generation of a minute sound wave by a sample that has absorbed electromagnetic radiation. The instrument tested in this study employs an inexpensive light-emitting diode to generate the signal and an inexpensive Radio Shack microphone to detect the signal. At the 660 nm wavelength employed in this study, the PA signal is directly proportional to the surface area of the carbon in the ash; thus, these measurements should correlate well with the Foam Index. If the sample is briefly ground to yield relatively uniform particle size, the PA signal will be proportional to volume (mass) of carbon; thus, these measurements should correlate well with accurate LOI determinations.

The instrument used in this study was developed at Iowa State University. A few modifications were made to the set-up, including the substitution of a light emitting diode (LED) for the original diode laser, substitution of an inexpensive Radio Shack electret microphone for the original wide dynamic range condenser microphone, and drilling a larger acoustical access hole between the PA cell and the microphone face.

PA signals were measured for fly ash samples both ground and unground. By grinding the sample to a reasonably uniform surface to volume ratio the signal then becomes proportional to the amount of carbon present. Unground samples, on the other hand, are expected to produced signals that are proportional to the total surface area of the carbon in the sample.

In previous studies with the PA instrument the PA signal from a fly ash sample was simply plotted against the carbon content as measured by organic carbon analysis.

However, in the present tests, the PA signal obtained from an as-received fly-ash sample was subtracted from the PA signal obtained from a fired sample of the same fly ash. This was done in an attempt to subtract out any background signal associated with fly ash in the sample. However, this procedure was, in a few instances, frustrated by fly ash that darkened upon firing, thus producing a higher signal from the fired samples than the as-received samples.

Organic carbon was determined by a commercial testing laboratory. All samples were first dried at 110 degrees C for one hour to remove moisture then a portion of the dried sample was washed in hydrochloric acid to dissolve inorganic carbon compounds. The original dried sample was then placed in a tall, alumina crucible and ignited at 700-800 degrees C. The total weight loss is reported as total carbon. The difference between the total carbon and the inorganic carbon is reported as organic carbon.

The Foam Index (FI) test is based upon a methodology described by Suuberg and coworkers. Eight grams of Portland cement and 2 grams of fly ash are placed in a 12-oz. Wide mouth jar. 25 ml of water is added and the jar shaken for one minute. From a stock solution of Daravair-1000 (W. R. Grace Inc.) (9 parts water plus one part Daravair-1000 by volume) drops are added from a dropper (1-5) at a time. Between each addition the jar is vigorously shaken for 15 seconds and the lid removed to examine the surface. The end point was declared reached when the foam remained on the surface for a minimum of 45 seconds. The total drops added are reported as surfactant taken up (adsorbed) by the sample.

The FI is plotted versus the PA carbon reading (unground) is relatively well correlated, with a regression coefficient R^2 equal to 0.85, as shown in Fig. 3. However, it appears that FI correlates slightly better (R^2 equal to 0.90) to organic carbon LOI than it does to the PA signal from unground fly ash. This result is a little surprising because organic carbon LOI is not expected to correlate to the surface area of the carbon.

With few exceptions, all three methods of analysis correlated to each other. The FI readings are strictly on a relative basis but the correlation at the higher carbon levels was 0.90 to the LOI analysis and some what less at 0.84 for the PA method. The correlation between the LOI and PA methods was again at 0.90. The method of firing fly ash samples for LOI analysis are thought to have produced reducing conditions that resulted in some samples darkening. As a result, some background PA measurements on fired samples produced anomalies that we think can be eliminated by improving the firing conditions during LOI tests.